

(19) World Intellectual Property  
Organization  
International Bureau



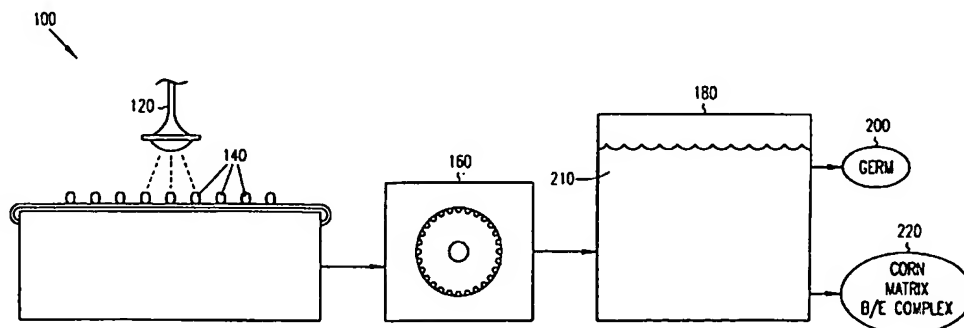
(43) International Publication Date  
18 August 2005 (18.08.2005)

PCT

(10) International Publication Number  
**WO 2005/074625 A2**

- (51) International Patent Classification: Not classified
- (21) International Application Number:  
PCT/US2005/003331
- (22) International Filing Date: 3 February 2005 (03.02.2005)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:  
60/521,001 3 February 2004 (03.02.2004) US  
10/975,195 28 October 2004 (28.10.2004) US
- (71) Applicant (for all designated States except US): BIOREFINING, INC. [US/US]; 4979 Olson Memorial Highway, Golden Valley, Minnesota 55422 (US).
- (72) Inventor; and
- (75) Inventor/Applicant (for US only): VAN THORRE, Doug [US/US]; 5251 Humboldt Avenue South, Minneapolis, Minnesota 55419 (US).
- (74) Agents: MCCrackin, Ann M. et al.; Schwegman, Lundberg, Woessner & Kluth, PA, P.O. Box 2938, Minneapolis, Minnesota 55402 (US).
- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Published:  
— without international search report and to be republished upon receipt of that report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: PROCESS FOR FRACTIONATING SEEDS OF CEREAL GRAINS



(57) Abstract: A method for separating bran, endosperm and germ in a seed particle, comprising: Smashing the seed against a surface that produces separation of the germ from a bran-endosperm complex; and Separating the germ from the bran endosperm complex.

### Process for Fractionating Seeds of Cereal Grains

5           This application claims priority to U.S. Provisional Application No. 60/521,001, filed February 3, 2004 and U.S. Application No. 10/975,195, filed October 28, 2004.

#### Copyright

10           A portion of the disclosure of this patent document contains material that is subject to copyright protection. The copyright owner has no objection to the facsimile reproduction by anyone of the patent document or the patent disclosure, as it appears in the Patent and Trademark Office patent files or records, but otherwise reserves all copyright rights whatsoever. The following  
15           notice applies to any software and data as described below and in the drawings that form a part of this document: Copyright 2005, Biorefining, Inc. All Rights Reserved.

#### Field

20           The invention described herein relates to a process for fractionating seeds of cereal grains and to fractionated cereal grain seeds.

#### Background

          Extraction and purification of biologically active materials from biomass  
25           has been a complicated and inefficient endeavor. Extraction has traditionally employed harsh solvents. The use of particular processing conditions and/or heat and/or chemicals have created intermediate reaction products and physical conditions very different from conditions forming the extracted chemicals in the first place. Because of these harsh conditions, there has been some question as  
30           to whether complex molecules such as native cellulose have ever really been extracted in a way that preserves the native cellulose structure.

          In the milling of cereal grains such as corn, wheat, and barley, a desired outcome of the milling is to segregate three major constituent parts of a cereal

kernel, which include germ, bran and endosperm. The milling industry has evolved around two processes: a wet and a dry process.

5 The wet milling process for corn includes steeping corn kernels in water, in which chemicals and heat have been added, for 24 to 48 hours, draining the water, recovering the kernels, coarsely grinding the steeped kernels and separating ground germ from a ground bran/endosperm complex by flotation. Next, the bran and endosperm are separated from the bran/endosperm complex. After separation, the three major constituents, the germ, the bran and the endosperm may be sold, as is, or may be further refined into value added  
10 products.

The dry milling process includes passing corn kernels through a hammer mill which chops the corn kernels into a mixture of small chopped pieces. The chopped mixture from the hammer mill contains all three constituent parts of the corn kernel. The dry milling process doesn't economically separate the three  
15 major constituents, germ, bran and endosperm, into their separate entities because the hammer mill output is not uniform. The dry milling process has been used almost exclusively to prepare the corn kernel constituents as a feedstock for the production of fuel ethanol by fermentation.

## 20 Description of the Drawings

FIG. 1 illustrates a schematic view of one process embodiment of the invention.

FIG. 2 illustrates a schematic view of one process for separating germ from a bran endosperm complex.

25 FIG. 3 illustrates a schematic view of separation of materials from a grain seed.

FIG. 4 illustrates a side view of a stator used in one process embodiment.

FIG. 5 illustrates a side view of a mixer-grinder-pump used in a process embodiment of the invention.

30 FIG. 6 illustrates an embodiment for separation of polymers, oligomers and monomers.

### Description

Embodiments described herein include a process for efficiently separating a cereal grain kernel into its constituents, one embodiment of which is illustrated at 10 in FIG. 1. A corn grain kernel includes constituents of germ, bran and an endosperm. The endosperm includes gluten and starch. The process embodiment 10 includes smashing corn kernels against a pestle, thereby forming a mixture of germ and a bran and endosperm, and ejecting the germ away from a complex of bran and endosperm, referred to herein as the bran/endosperm (b/e) complex. This smashing is also referred to as impact milling. One impact milling machine usable in the process embodiment is manufactured by Entoleter of Handen, Connecticut.

No heat or chemicals are required in order to perform impact milling. Furthermore, with impact milling, the corn germ retains its physical dimensions. With impact milling and additional processing, the corn germ is impelled in order to recover corn oil more efficiently.

Additionally, the corn germ particles are reduced to produce a more consistent, monodispersed material. By "monodispersed" is meant that the processed particles are substantially the same size. Additionally, impact milling of a cereal grain and a germ component of the grain reduces processing problems related to differential hydrolysis that occur in wet milling.

Once the kernel is impacted and the germ, and a bran/endosperm complex are separated from the kernel, the germ, and bran/ endosperm, also referred to herein as the b/e complex, fractions are separated from each other. In one embodiment, to separate the corn germ from the bran/endosperm complex, a hypertonic solution is prepared in a vessel that then is used to float the germ away from the b/e complex. While a hypertonic solution is described, it is understood that other methods are usable to separate the corn germ from the b/e complex.

The aqueous hypertonic solution has a density and a volume effective for floating the corn germ and sinking the bran endosperm complex. The floating germ is skimmed at or near the top of the solution level in the vessel. The remaining corn material is extracted at or near the bottom of the vessel.

In one embodiment, one or more of the impact milling and hypertonic separation is performed as a continuous process. In one embodiment, the hypertonic solution is prepared using a material that is usable in other operations of the process. For instance, fermentable sugar is usable to prepare the hypertonic solution. Once the fermentable sugar is used as a separating hypertonic solution, the spent solution is usable in other operations of the overall process. For instance, the spent hypertonic solution that includes fermentable sugar is also usable as feedstock to a fermentor. This embodiment of a corn germ separation process is then a continuous, closed loop, zero discharge process.

For some embodiments, the b/e complex is subjected to a cryogenic processing tank filled with liquid carbon dioxide that instantaneously freezes the b/e complex. One embodiment of a cryogenic tank is described in U.S. Pat. No. 4,886,534, which issued December 12, 1989. This tank is presented for exemplary purposes only and is not meant to limit embodiments of the invention described herein. Because there is differential vapor pressure between the bran and the endosperm that form the bran endosperm complex, the junction where the bran and endosperm meet weakens and separates. The b/e complex is then passed through a pair of rollers that finalizes the separation of the bran from the endosperm.

Because carbon dioxide is inert, it leaves no residue on the grain fraction particles and may be recycled. With this process, the bran retains its physical shape while the endosperm is shattered into smaller pieces.

For some embodiments, centrifugation is used to separate the bran from the endosperm. The separated bran is of uniform physical size and chemical makeup, i.e. pentose sugars. For some embodiments, the endosperm is then finely ground in a roller mill and the starch and corn gluten (protein) are separated by centrifuging. In one embodiment, the starch is further processed to a desired end product. The separated corn gluten, now called zein, is segregated and is sold as is or further processed.

Another embodiment of the invention described herein, illustrated schematically at 100 in FIG. 2 includes a wetting device 120 for wetting kernels, one kernel of which is at 140, prior to impact milling the kernels; an impact mill 160 for performing impact milling and a vessel 180 for containing the hypertonic

solution 210 and for separating corn germ 200 from the remaining corn matrix 220. For some embodiments, the wetting device 120 is used in conjunction with one or more dry mills.

One suitable kernel wetting device includes a nozzle capable of  
5 delivering water as a spray to kernels that are on a moving bed or are in a hopper. While a kernel wetting device is described for one embodiment, it is understood that other system and method embodiments do not include a wetting device. Instead, the kernel is impacted while dry. The kernel milling is performed using one or more impact mills, such as those described above. The  
10 corn kernels are wetted prior to milling to a degree sufficient to transport the kernels to and through a mill and to reduce the amount of corn particle dust generated and to reduce or eliminate requirements for dust collection equipment. The wetting also helps in separating the germ from the bran/endosperm complex.

The germ separation is performed in an impact milling process  
15 embodiment of the invention. Impact milling does not damage the pericarp and endosperm. Consequently, these materials are capable of being subsequently processed into value added products.

The corn germ contains approximately 85% of the oil found in the virgin corn kernel. Separating the corn germ improves the extraction and recovery of  
20 the oil from the germ and the processing of protein in the germ. Furthermore, the corn oil is not present in subsequent process steps to interfere or complicate the further processing of the corn kernel complex which is generally made up of the pericarp and starch.

The process embodiments described herein separate the corn germ  
25 cleanly and do not damage the pericarp and endosperm. As a consequence, the pericarp and endosperm are optionally processed into value added products. Some of the process embodiments of the invention use dry corn germ separators, milling devices, which have a long history of performing relatively well in separating the corn germ from the pericarp/endosperm complex.

30 Once the germ and pericarp are separated from the pericarp/endosperm complex, the endosperm is subjected to size reduction and filtering to separate the gluten. One method and system embodiment uses a high-frequency, rotor-stator shearing device in the treatment of endosperm. This type of device produces high-shear, microcavitation forces which defibrillate the endosperm fed

into it. Two commercially available high-frequency, rotor-stator dispersion devices are the Supraton™ devices manufactured by Krupp Industrietechnik GmbH and marketed by Dorr-Oliver Deutschland GmbH of Connecticut, and the Dispax™ devices manufactured and marketed by Ika-Works, Inc. of Cincinnati,  
5 Ohio. These devices are mentioned to provide examples of suitable devices but are not meant to limit acceptable high-frequency, rotor-stator shearing devices usable in the processes of the present invention.

To prepare the endosperm and any remaining pericarp for shearing, the endosperm is first reduced to a manageable size by grinding. Grinding to a  
10 desired particle size is accomplished in one or more stages. In a general aspect of the process, the milled endosperm/pericarp is ground by conventional hammer milling to a particle size sufficiently small enough to pass through a number 4 mesh sieve.

In one embodiment, the ground product is mixed with water to obtain a  
15 slurry of a preselected solids content. One of the purposes of this part of the process is to swell and further to defibrillate the starch. In one embodiment, the ground starch is fed into a hopper and is conveyed to a mixer-grinder-pump. Water is added to form a slurry having a solids content ranging from about 10% to about 25% solids. In one embodiment, the mixer-grinder-pump is a medium  
20 shear, rotor-stator device capable of mixing and pumping high solid content slurries. This device further reduces the particle size of the starch, wets the particles thoroughly with water, and disperses the particles within the water. Examples of this type of device are the HED™ manufactured and marketed by Ika Works, Inc. of Cincinnati, Ohio and the Gorator™ manufactured by  
25 Krupp Industrietechnik GmbH and marketed by Dorr-Oliver Deutschland GmbH of Connecticut.

Referring to FIG. 4, a starch slurry is fed into the high-frequency, rotor-stator device 111 and is forced into a chamber 110. Inside the chamber is a series of coaxial meshing rings. The rings are configured with teeth, slots or bore holes.  
30 The rings configured with teeth are generally known as tooth and chamber tools and those configured with bore holes are generally known as nozzle tools. Generally, tooth and chamber tools are attached to both the rotor and the stator when tooth and chamber tools are used. When nozzle tools are used, generally, a

tooth and chamber type tool is affixed to the rotor and a nozzle tool will be affixed to the stator.

The rings are concentric, radiating out from the center. The rings 112 on the stator are fixed and the rings 114 on the rotor are rotated by a shaft coupled to a motor.

The structure identified as 116 is representative of a tooth on a tooth and chamber tool attached to the rotor. The structure identified as 118 is representative of both a tooth on a tooth and chamber tool attached to the stator and the body of a nozzle tool spaced between bore holes. Accordingly, the space identified as 122 represents the gap between the teeth on a tooth and chamber tool attached to the rotor. The space identified as 120 represents both the gap between teeth on the stator tooth and chamber tool attached to the stator and the gap formed by a bore hole in a nozzle tool attached to the stator. The rings 114 on the rotor and the rings 112 on the stator are closely spaced at close tolerances. The space between the rotor and stator is typically about 1 mm.

Regarding a tooth and chamber tool, adjacent pairs of teeth are separated by gaps 120 and 122. The tooth and gap size determine the coarseness of the machine, i.e., a coarse tool has fewer teeth with larger gaps between adjacent teeth when compared with a medium or fine tool. Both the Supraton™ and Dispax™ allow the use of coarse, medium, and fine toothed rings in the same device, or the devices can have all coarse, all medium, or all fine toothed rings in the chamber so that the machines may be used in series, if desired. The use of multiple devices in series is one alternative to the use of a single device for processing starch.

As the starch slurry is pumped under pressure into the chamber 100 by the mixer-grinder-pump, the slurry encounters each concentric layer of the tools in place in the chamber as the slurry is forced laterally. This lateral force is created by the pressure on the slurry as it is pumped into the chamber by the mixer-grinder-pump and by the centrifugal force created by the spinning rotor. The slurry passes through the gaps between the teeth as the rotor spins past the gaps in the stator. Flow is most pronounced when the gaps 122 between the rotor teeth align with the gaps 120 in the stator. The result is a pulsing flow with a rapid succession of compressive and decompressive forces. The starch material in the slurry is subjected to these repeated forces, as the centrifugal force



accelerates it through the gaps toward the outer edge of the chamber. As the slurry moves towards the outer edge of chamber 110 the centrifugal forces increases, thus intensifying the forces generated in gaps 120 and 122. The repeated compressive and decompressive forces create microcavities in the slurry with extremely intensive energy zones. These zones are illustrated at 402, 404, 406 and 408 in Figure 5. The starch structures are ripped apart by these forces. Additionally, the resulting starch structures exhibit extensive internal decrystallization due to the forces generated in the microcavities.

As the starch particles pass outward through the various gaps, the particles come in contact with the teeth and the body of the nozzle tool. Accordingly, some grinding of the particles may occur due to such contact. The grinding effects are relatively small, however, when compared with the combined effects of shear forces and microcavitation. Nonetheless, as solids loadings increase the instance of grinding may also increase.

Grinding typically cuts, slices, and dices fibrous material perpendicular to the fiber bundle, producing a more spherical type of particle. Shear forces in combination with microcavitation, on the other hand, tend to shatter the material, that is, they rip the fibers apart from the inside-out explosively forming irregularly shaped particles. Examination of these particles show them to have been "cut" both perpendicular to the fiber axis and longitudinally along the fiber axis. The effect on the starch particles is to shatter their structure, possibly disrupting the starch bonding without the compressive effects of grinding. Solids loadings not exceeding 30% are employed to minimize grinding of the starch and thus the compressive effects of the grinding.

While the precise mechanisms occurring within the chamber of the high-frequency, rotor-stator device are not totally understood several factors are thought to aid in explaining the effects on the treated starch. The swelling effect of liquids, particularly water, is thought to aid in creation of longitudinal shearing effects in the treated starch. The repeated compressive and decompressive events in and between the gaps are thought to create internal pressures tending to explode the starch particles and thus the starch structure thereof. It is also hypothesized that a harmonic resonance effect may be created during operation of the rotor-stator device in the sonic range. Thus, a harmonic frequency of a particular length when reached during processing would cause the

effected particles to resonate and tend to aid in the destruction of the particle structure of the starch and any remaining pericarp.

As previously stated, high-frequency, rotor-stator dispersion devices may have differently configured rings or "tools" within the chamber. These tools, for example, may vary in the gap size between the teeth on the rings or in bore hole size in the case of a nozzle tool. With a larger gap size, the resulting material is more coarse than with a smaller gap size. As stated earlier, these tools can be varied within one device to contain coarse, medium, and fine rings in the chamber of the device. Likewise, a device may contain rings of the same rating so that the devices can be staged. This capability is important for use in a continuous process.

Once the starch is treated by a high-frequency, rotor-stator dispersion device, the treated starch is optionally subjected to pressurization and depressurization. The process of pressurization and depressurization with saturated steam forms hydrolysates—from hydrolysate fractions having the highest water content to hydrolysate fractions having the lowest water content. Once extracted, the fractions are cooled to ambient temperature. For some embodiments, the fractions are kept hot for further processing. With this method, there is a minimal loss in biofunctionality and bioresponse, as compared to traditional wet chemistry methods of separation. What is remarkable and unexpected is that this biofunctionality and bioresponse is achieved without complicated chemical treatment. Separation without loss of functionality and response is achieved by a one step steam pressurization/depressurization. What is also remarkable is that the extraction occurs with virtually any biomass feedstock. Pretreatment of biomass is minimal and is typically limited to size reduction.

The starch is then subjected to fermentation using conventional fermentation equipment and procedures or further processing to glucose.

For some embodiments, fractions obtained from fermentation are then subjected to ion exchange for the final purification.

It is believed that with this treatment, substantially native physical and chemical properties and structure are preserved for molecules such as native cellulose. It is also believed that with this treatment, a mass balance can be performed over a biomass for virtually all of the bio-functional materials within

the biomass. Products extracted are in a concentration and having a reactivity within a range of what is predictable from a mass balance.

Embodiments of the invention described herein also include a process for extracting, separating, and purifying individual stereoisomers and other specialty chemicals from components such as the germ or the pericarp. One process, illustrated schematically in Fig. 3 and at 1000 in Fig. 6, comprises providing a source of biomass 1200, such as corn germ after oil has been extracted and the pericarp, and subjecting the biomass to saturated steam pressurization/depressurization 1400 that increases surface area of the biomass and that permits separation of lignin, cellulose, and hemicellulose components from the biomass, heating hemicellulose 1600 separated from the biomass in order to hydrolyze the hemicellulose and obtain hydrolyzed monomers, oligomers and polymers, and separating polymers, oligomers, and monomers from hydrolyzed hemicellulose 1800. While hydrolysates are described, it is understood that the process of the present invention is usable to extract, separate and purify constituents and derivatives of cellulose, hemicellulose and lignin. For instance, cellulose derivatives such as carboxy methyl cellulose and hydroxypropyl cellulose can be obtained using the process of the present invention. Coniferyl alcohols are also obtainable from the lignin. Stereoisomers of the monomers are further extracted using chromatographic methods 200.

Embodiments of the invention achieve high yields of stereoisomers, when present, such as L-arabinose, using physical processes in addition to hydrolytic reactions, rather than exclusively conventional, water based, chemical extraction techniques. It has surprisingly been found that employing heat and pressure in treating biomass, such as corn germ and pericarp, increases production rates and percent yield of stereoisomers as compared to conventional, water based, chemical extraction processes.

As used herein, "simple sugars" refer to monosaccharides and oligosaccharides which are not decomposed into degraded products upon hydrolysis. Monosaccharides include pyranoses and furanoses. Monosaccharides are also classified according to the number of carbons in the molecule; for example, glucose is a hextose.

As used herein, "complex sugars" refer to polysaccharides which are carbohydrates of high molecular weight capable of being hydrolyzed into a large

number of monosaccharide units. Typical polysaccharides are cellulose, lignin, hemicellulose, starch and pentosan.

An oligosaccharide is a simple polysaccharide with a known number of constituent monosaccharide units, such as 1 to 10 monomers.

5           The term "biomass" as used herein refers to plant materials including, but not limited to corn germ and pericarp. Biomass in the form of plant materials includes cellulose and hemicellulose, both of which are polysaccharide, and lignin. Native cellulose molecules are linear and unbranched glucose polymers with a high degree of polymerization (DP) between 3500 to 12,000 and 12,000  
10 to  $10^6$  DU. Cellulose has a strong propensity to form both intermolecular and intramolecular hydrogen bonds. Cellulose is stable against degradation under most physical and chemical conditions. Hemicellulose comprises heteropolysaccharides which are formed by a variety of different monomers. Most commonly the monomers are glucose, galactose, mannose, xylose and  
15 arabinose. Hemicellulose molecules have a degree of polymerization of about 10-1000. The term "feedstock" as used herein refers to any material supplied to a device, machine, or processing plant.

The biomass is subjected to a particle size of reduction to a size of chips or finer, such as a size of sawdust, using conventional particle reduction  
20 equipment. The smaller the size, the easier it is to mechanically handle the biomass. Smaller sized particles have a greater surface area and are more amenable to chemical reaction, such as solubilization. Also, desired processing temperatures are reached more rapidly when using smaller particles.

In one embodiment, the biomass is fed into the high-frequency, rotor-stator device, illustrated schematically at 110 in Figure 2 and is forced into a chamber 110, shown in Figure 4. Inside the chamber is a series of coaxial meshing rings. The biomass treatment in the high-frequency, rotor-stator device is described above.

In one embodiment, the biomass is fed to a hopper following treatment in  
30 the rotor-stator device. The biomass may optionally be sprayed with water either before transfer to the hopper or while in the hopper. The biomass exits from the bottom of the hopper into a conveying feeder which contains a conveying mechanism such as a feed screw driven by a variable feed drive. The feed screw or other conveying mechanism feeds the material into a compacting feed tube

and then into a pressurized retention tube, where the biomass particles are formed into a solid plug of material. The solid plug is compressed by surface pressures of up to 2000 psi.

5 The biomass is mechanically compacted prior to its introduction into the digester. The biomass is desirably in a moistened condition. The mechanical compaction removes air from the material prior to its introduction to steam pressurization. Air is undesirable because oxygen in the air tends to oxidatively degrade the biomass. Air also exerts a partial pressure and retards temperature and pressure equalization within the reactor.

10 Steam pressurization, within the pressurized reaction vessel, is typically operated with automatic pressure and temperature control systems. The partial pressure of any air pockets decreases steam pressure and temperature in the reactor below a preselected value. Compaction, followed by processing conditions discussed below, causes a degree of water saturation of the biomass.

15 Water saturation of biomass assists in the heat transfer within and around the material.

Next, the biomass amorphous fibers are loosened by steam pressure treatment and defibrination. In particular, the particles are treated with saturated steam at a temperature of from about 160 to 230 degrees Centigrade for a period

20 of time from 2 minutes to 4 hours. The biomass is disintegrated by this steam treatment. In general, the lower the temperature used, the longer the duration of treatment should be. Thus, for some extractions, it is desirable to treat a biomass at 160 degrees Centigrade for about 4 hours. For other extractions, it is desirable to treat a biomass for 2 minutes at 230 degrees Centigrade.

25 This steam treatment helps separate fractions within biomass by most to least water content. The fractions are separated as extractables such as terpenes, fatty acids and so forth, lignin, pectin, hemicellulose and native cellulose. This steam treatment yields fractions at yields that are predictable by a mass balance of the biomass. In other words, the steam treatment and extraction of the present

30 invention permits a user to ascertain bioactive/biofunctional materials present in living biomass and to extract the bioactive/biofunctional materials in quantities that approach or are substantially the same as the materials are present in the native biomass.

Biomass disintegrated this way is then, subsequently, for some embodiments, lixiviated with an aqueous solution of alkali. The concentration of NaOH is typically no greater than about 4% by weight.

In another embodiment the biomass mixture contains between 2 and 50  
5 grams of alkali, hydroxide of sodium or hydroxide of potassium, per 100 grams of dry biomass.

The steam pressure treatment is performed in either a continuous stream or a batch type steam pressure reactor. In one embodiment, the reactor is manufactured by Stake Technology Ltd. Of Ottawa, Canada. One particular  
10 device is described in U.S. Pat. No. 4,136,207, which issued January 23, 1979, and which is herein incorporated by reference. The steam pressure treatment is performed in the reactor vessel. The reactor vessel is maintained at a pressure that is between about 200 and 450 psig. The temperature in the reactor is maintained between about 390°F and 460°F. The biomass is fed intermittently  
15 for some embodiments and continuously for other embodiments. By varying the biomass stream but maintaining the reactor vessel conditions, the method of the present invention introduces an efficiency to the process, by avoiding ramp up and ramp down conditions within the reactor vessel.

The biomass is introduced into the reaction vessel in a manner that forms  
20 a solid plug at the inlet of the vessel. In one embodiment, the solid plug is formed in a device, such as a retention tube. The biomass plug prevents a loss of pressurization in the vessel. The combination of the biomass plug and constant pressurization permits instantaneous steam penetration of the biomass within the reaction vessel, and thus permits better control of processing times.

25 The biomass is processed at the steam temperatures described for a period of at least about 15 seconds and for some embodiments, at least about 5 minutes. The maximum time is about one hour.

After cooking, the biomass is cooled and depressurized substantially  
instantaneously. The biomass is in a moisture saturated condition. The biomass  
30 is subjected to sudden and substantially instantaneous decompression and adiabatic expansion, e.g. by discharging a small quantity of cooked biomass into ambient conditions.

The process of instantaneous pressurization and de-pressurization separates the biomass into components of lignin, cellulose and hemicellulose.

The hemicellulose product is separated from the cellulose product and lignin product by techniques known in the art. It is further contemplated that the cellulose product is separated from the lignin product by techniques in the art.

Once the hemicellulose is extracted from the biomass, the hemicellulose  
5 for some embodiments, is heated in a steam heater, such as a Komax steam heater and then is hydrolyzed in a static mixer, such as a Komax reactor/static mixer, manufactured by Komax Systems, Inc., of Long Beach, CA. One reactor/static mixer embodiment is described in U.S. Patent No. 6,027,241. The reactor/static mixer is, in one embodiment, constructed so that an additive, such  
10 as sodium hydroxide is added countercurrent to the main fluid stream. The heater and mixer comprise a heater-mixer system.

Within the reactor, at approximately 329°F hemicellulose undergoes a phase transition, depending upon the moisture content, from a solid to a non-Newtonian fluid, somewhat like tooth paste. At temperatures higher than  
15 approximately 500°F, depending upon moisture content, the hemicellulose begins to pyrolyze. Furthermore, the xylan component of the hemicellulose is degraded at temperatures above 428°F. Hence, to preserve the quality of the hemicellulose product stream, the hemicellulose exposure to temperatures above 356°F should be as short as possible. The in-line reactor heater--static mixer  
20 system raises the temperature of the hemicellulose to between 329°F and 347°F. The time to bring the temperature within this range is typically less than about 10 seconds to about 20 seconds.

Once heated, the hemicellulose is reacted with NaOH in the reactor/static mixer. The static mixer accepts the hemicellulose, a high viscosity stream and  
25 NaOH, the low viscosity stream. The NaOH is injected into the high viscosity stream, mixed by static mixing and a chemical reaction occurs between the alkali and the hemicellulose. In particular, the NaOH hydrolyzes the hemicellulose. The process of the present invention, unlike conventional sugar extraction processes, does not rely upon chemical reactions for extraction. Instead, the  
30 process of the present invention utilizes both sophisticated mechanical separation, occurring in the static mixer, coupled with NaOH addition for hydrolysis, for extraction and formation of hydrolysates.

In one embodiment, the hydrolysates include dissolved solids at a concentration of about four weight percent. The specific cation analysis

included magnesium in a concentration of 2.5 ppm; calcium in a concentration of 3.66 ppm; potassium in a concentration of 0.40 ppm and sodium in a concentration of 1.81 ppm. The hydrolysate is prepared at 205 degrees Centigrade for three minutes with no acid.

5           The oligomers in the hydrolysates are converted to monomers without the addition of acid and are separated, in some embodiments, by ion exchange. In one embodiment, ion exchange columns are one inch in diameter and contain resin that is 3-4 feet deep. Ion exchange beds include a first cation resin bed and a second bed. Ion exchange beads are styrene crosslinked with divinyl benzene.  
10       Flow through the ion exchange bed is at 60 to 80 degrees Centigrade and is retained in the bed for a fifteen minute retention time. There is then a total retention time of thirty minutes through both of the beds. The conversion of the oligomers to monomers in the hydrolysate is then measured. If conversion not at a preselected value, the hydrolysate effluent is heated to 80 degrees Centigrade  
15       and samples are taken until conversion is completed. In one embodiment, the liquor provided to the ion exchange beds is a mixture of oligomers (3/5) and monomers (2/5).

          If acidity is insufficient to complete the conversion, acid is added to the cation effluent to drive the reaction. The cation effluent pH is typically within a  
20       range of 2.5 to 3.5. Post hydrolysis is performed mildly with acid and once oligomers are converted to monomers, the reaction mixture is concentrated by vacuum evaporation to 50 percent dissolved solids.

          With the process of the present invention, the hydrolysate mixture is further hydrolyzed to the basic monomeric unit from oligomers, and polymers in  
25       a single step and then separated on the basis of stereoisomer, i.e. optical or chirally pure monomer separation, in a second step. In another embodiment, the hydrolysate mixture is separated and a desired stereoisomer may be extracted in a single step.

          In one embodiment of the process of the present invention, sugar  
30       products obtained by ion exchange are crystallized. In one embodiment, the crystallization is performed using a low intensity ultrasonic agitation. It is believed that this crystallization produces a product wherein crystals have few inclusions, are uniform in shape, in size, in density and in purity.



In one embodiment, the L-arabinose is separated from other monomers using the ion exchange methods and resin described herein. In another embodiment, the L-arabinose is separated from a mixture of hemicellulose hydrolysates.

5           For some embodiments, the high-frequency, rotor-stator treatment is used in a process that includes the ion exchange embodiment. For other embodiments, the process includes one of either the high frequency, rotor-stator treatment or the ion exchange embodiment.

          Further, the process embodiments produce high yields of grain  
10 components because, during recovery and processing, material isn't lost due to particles that have been too finely ground. Also, there is no solubilization of kernel constituents in water.

          With process embodiments of the invention described herein, materials are processed to a uniform physical size and chemical profile. All kernel  
15 components are recovered in pristine condition and therefore retain inherent functionalities. Embodiments of the process are adaptable to any cereal grain.

          Prior art processes, such as wet milling, require a use of water, in order to steep the corn and then ferment corn fractions in a fermentator. Water is also used to dissolve soluble fractions from the corn. This use of water adds to the  
20 difficulty of recovery and decreases the yields and efficiencies of processing the corn economically. Embodiments of the invention do not require water in quantities required for wet milling.

          Embodiments of the invention described herein replace the grinding or hammer milling of a kernel with impact grinding. Since conventional grinding  
25 and milling causes a range of physical sizes (polydispersion) of a kernel to be produced, further processing is compromised because the size distribution of particles causes uneven processing to occur by having under processed or over processed material as well material with the desired amount of processing.

          The present invention may be embodied in other specific forms without  
30 departing from its spirit or essential characteristics. The described embodiments are to be considered in all respects only as illustrative and not restrictive. The scope of the invention is, therefore, limited only by the appended claims rather than by the foregoing description. All changes which come within the meaning

and range of functional equivalency of the claims are to be embraced within their scope.

**CLAIMS**

1. A method for separating bran, endosperm and germ in a seed particle,  
comprising: Smashing the seed against a surface that produces separation of the  
germ from a bran-endosperm complex; and Separating the germ from the bran  
5 endosperm complex.
2. The method of claim 1, further comprising freezing the bran endosperm  
complex.
- 10 3. The method of claim 1, further comprising separating the bran from the  
endosperm intact.
4. The method of claim 2 wherein the bran endosperm complex is frozen  
with liquid carbon dioxide.
- 15 5. The method of claim 2 wherein the force is applied with a roller mill.
6. The method of claim 1, further comprising separating the bran from the  
endosperm.
- 20 7. The method of claim 5 wherein the separation includes centrifugation.
8. The method of claim 1 further comprising separating bran pentose  
sugars from the endosperm.
- 25 9. The method of claim 1, further comprising separating starch and protein  
from the endosperm.
10. The method of claim 1 wherein the seed is a corn kernel.
- 30 11. The method of claim 1 wherein the surface separates germ from the bran  
endosperm complex without evolution of heat and without water.

12. A bran particle produced by the method of claim 1 characterized by having a symmetry substantially unchanged from its native symmetry.
13. A bran particle produced by the method of claim 1.
- 5 14. A germ particle produced by the method of claim 1.
15. A system for separating bran, endosperm and germ from a seed, comprising: A unit having a surface that produces separation of germ from bran  
10 endosperm complex in a seed, without evolution of heat.
16. The system of claim 15, further comprising a unit for freezing the bran endosperm complex.
- 15 17. The system of claim 15, further comprising a unit for crushing the endosperm while separating the bran intact.
18. A germ separated from an oil-bearing seed, according to the method of claim 1, the germ retaining substantially all of the oil held by the germ when it  
20 was part of the oil bearing seed.
19. A system, comprising: an impact mill; a vessel and an hypertonic solution contained in the vessel; and a cryogenic tank.
- 25 20. The system of claim 19, further comprising a water dispersing device.

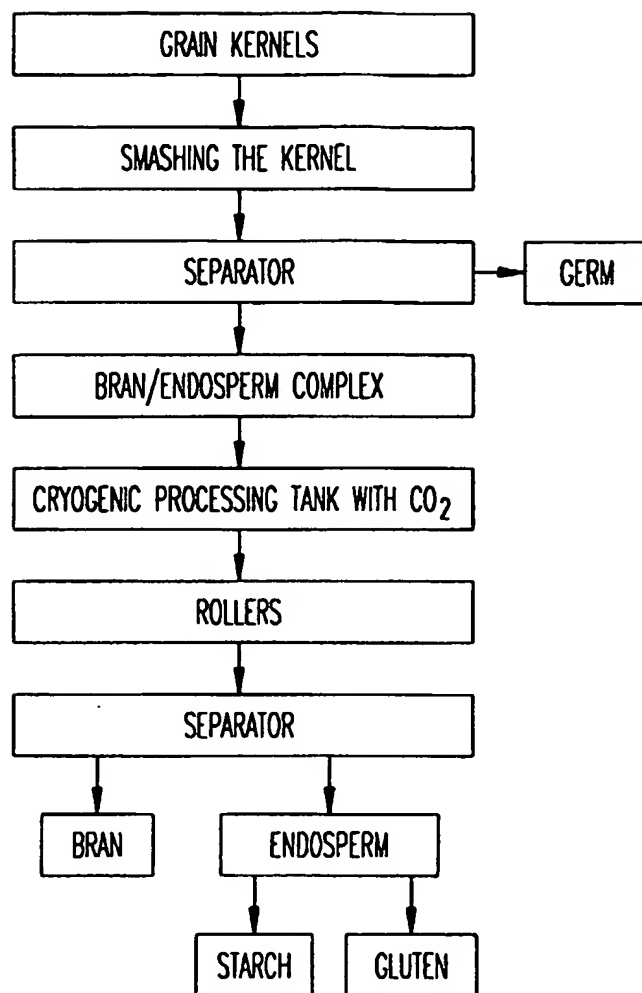


FIG. 1

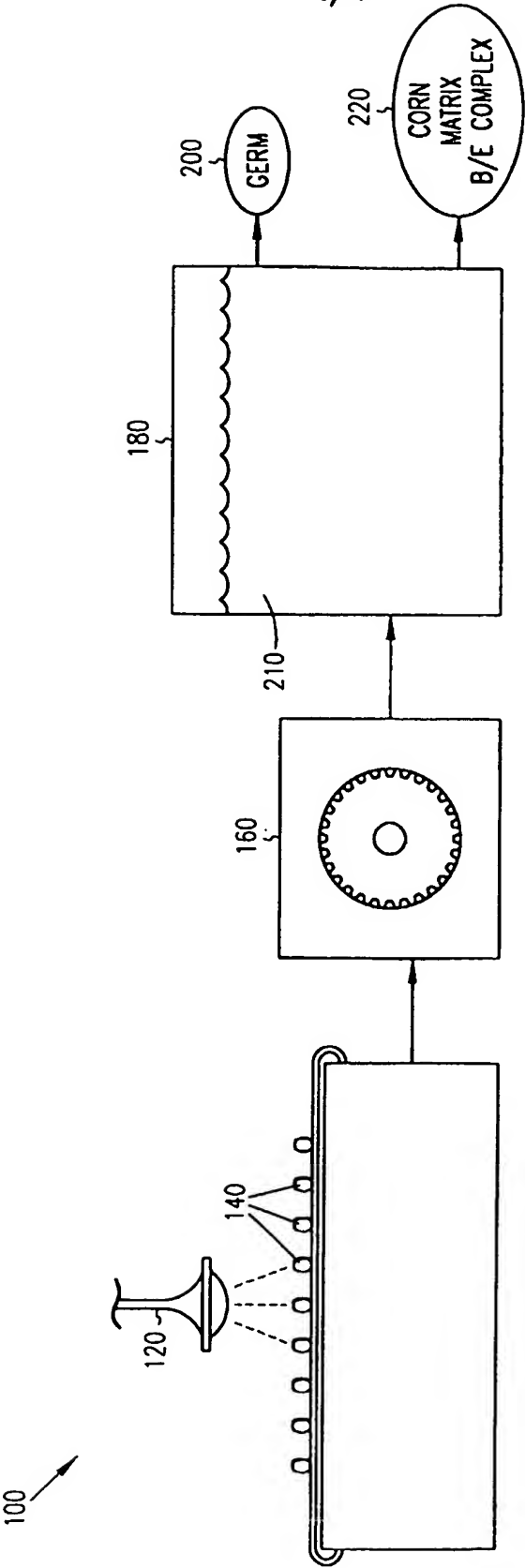
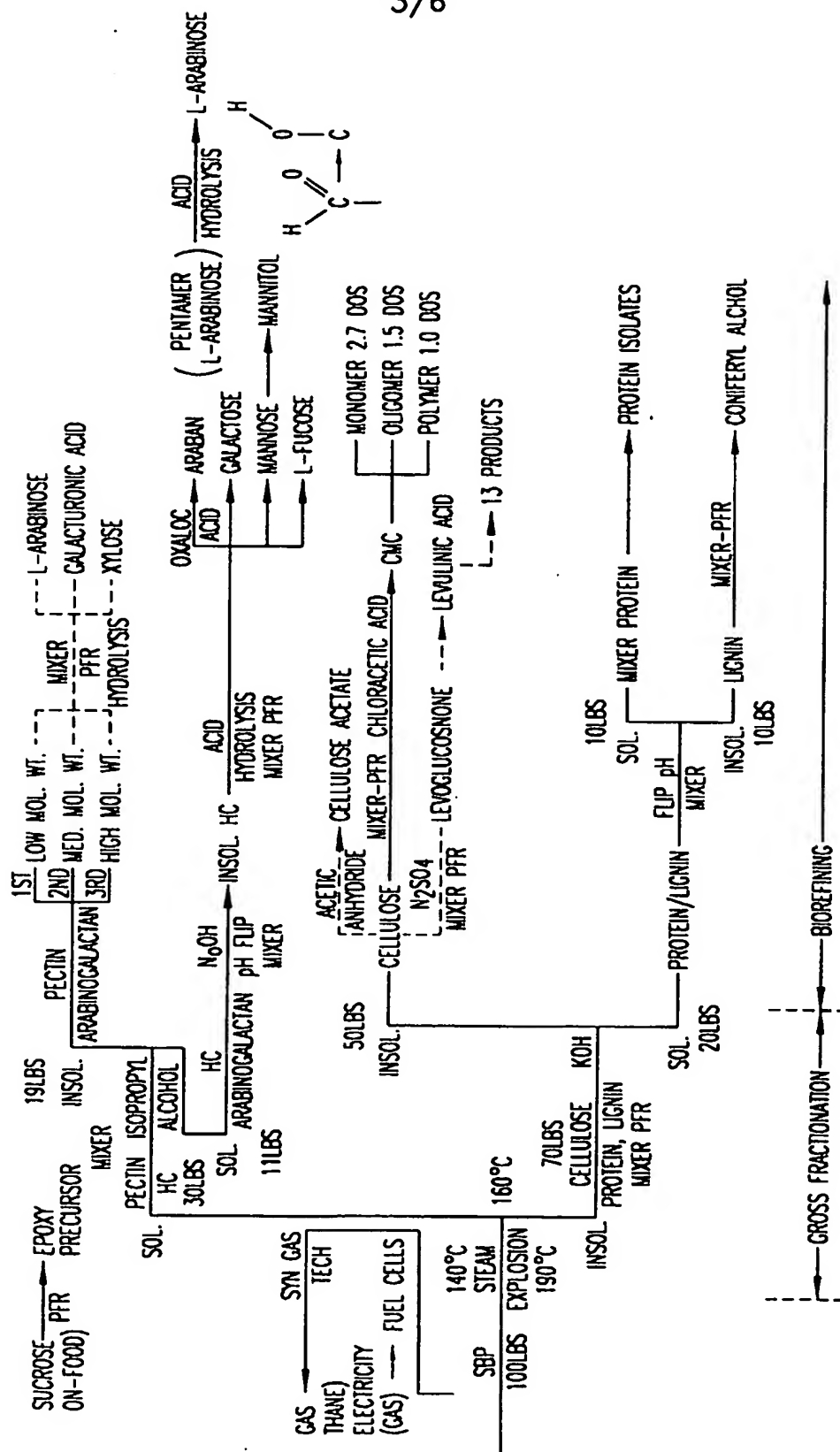


FIG. 2



**FIG. 3**

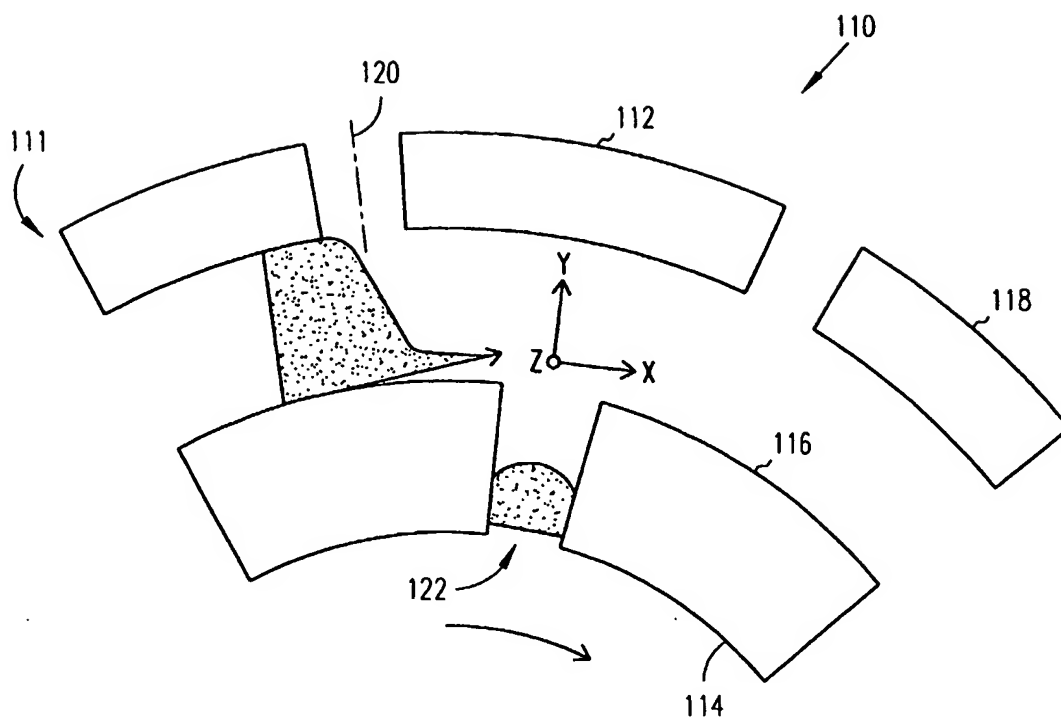


FIG. 4



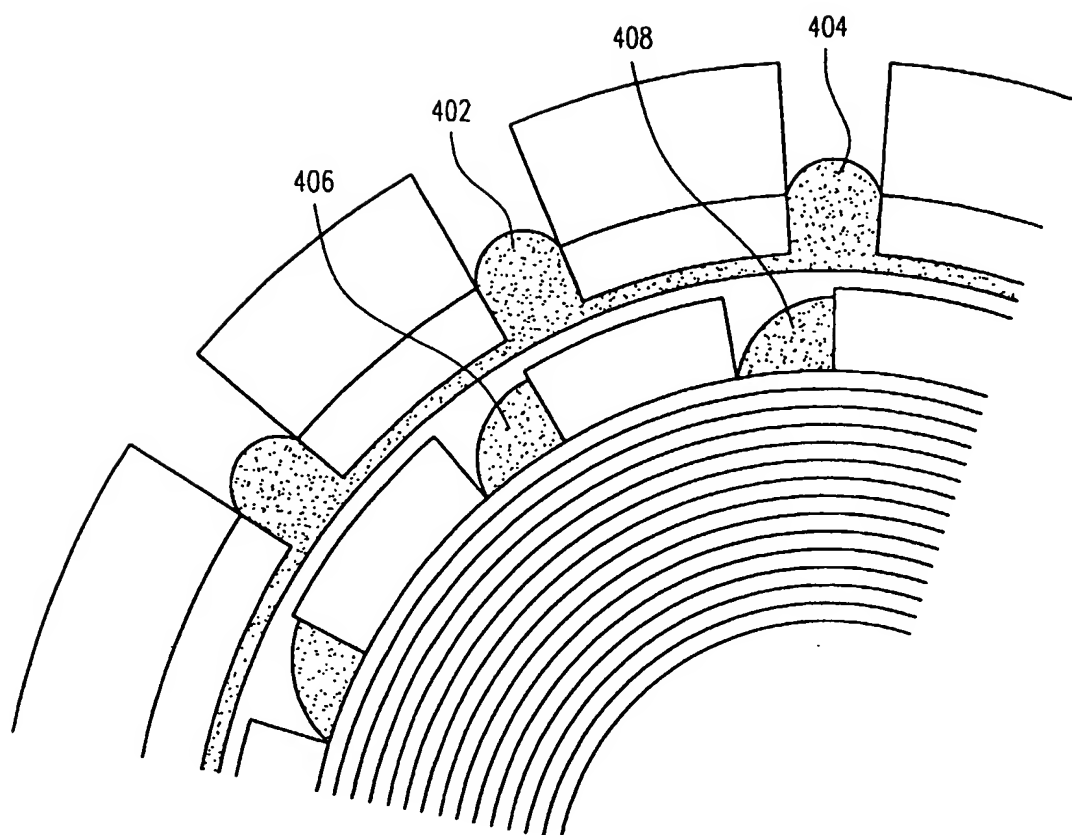


FIG. 5

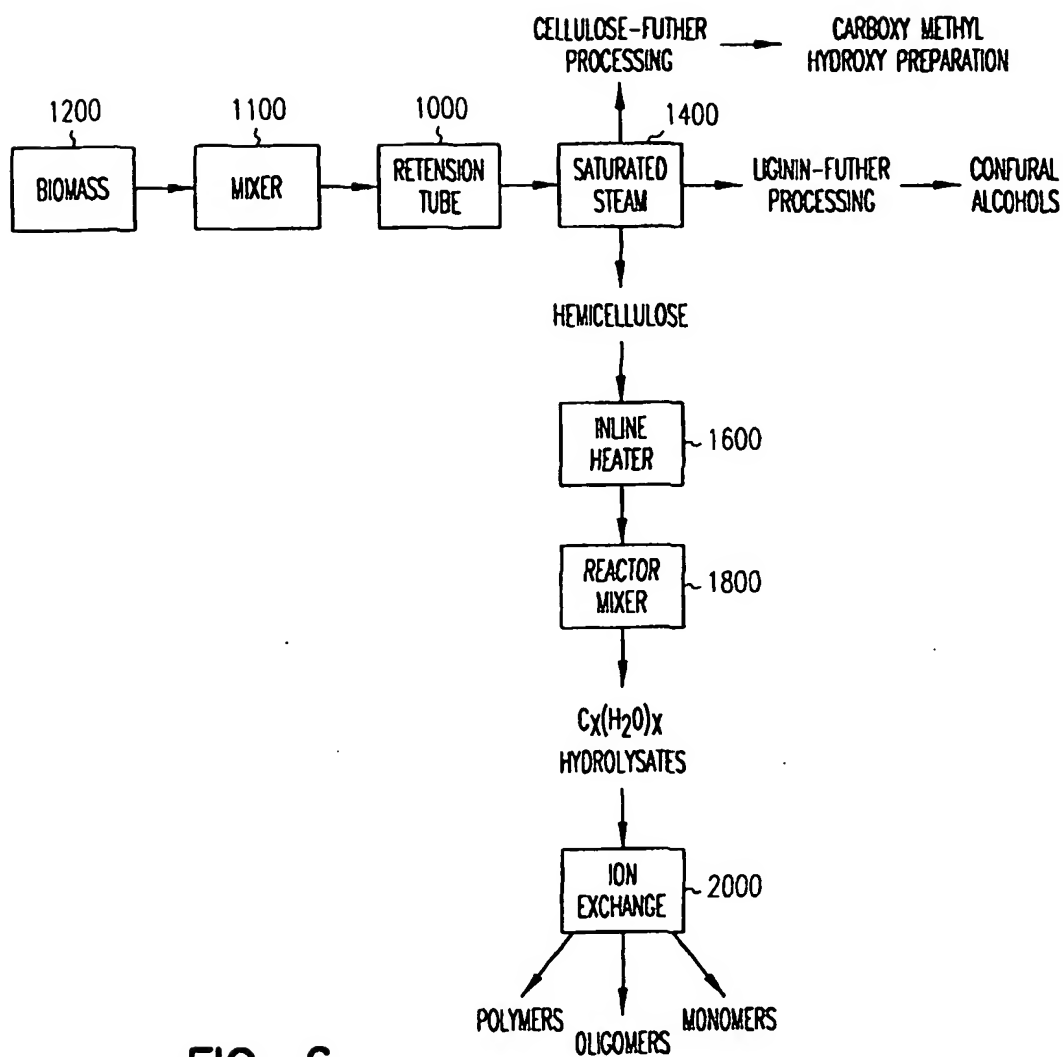


FIG. 6